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For: HAND-HELD MICROWAVE INTRA-ORAL DENTAL SYSTEM

SUBMISSION OF PRIORITY DOCUMENT

Assistant Commissioner for Patents
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Sir:

Enclosed is a certified copy of the Canadian patent application number 2,246,663 to which the above-identified U.S. patent application corresponds.

Respectfully submitted,

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Specification and Drawings, as originally filed, with Application for Patent Serial No: **2,246,663**, on September 18, 1998, by **MARC M.S. SEGHATOL**, for "Dental Microwave Application Method, Apparatus and Polymer Based Compositions".

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Dental microwave application method, apparatus and polymer based compositions

Objects containing or consisting of polymers are generally used in the dental arts for the restoration of lost tissue and the improvement of oral functions. They are required to have specific physical, mechanical, chemical and biological properties, for example composite fillings or dentures should have adequate strength, durability, processing and dimensional accuracy. They should be highly and appropriately polymerized to improve strength and stability, and they should be biocompatible and chemically inert. They should be able to be processed rapidly and conveniently. An example of a polymer object used in the dental arts is a removable denture. Most commercial dentures consist of a methyl methacrylate polymer, a copolymer, a methyl methacrylate monomer, a crosslinking agent, an initiator, an accelerator and other additives. The denture base may be cured by water-bath, microwave heating or light. Thermal water-bath curing requires up to 8 hours for processing flasked denture since plaster molds and polymers are poor thermal conductors. Furthermore, due to polymerization shrinkage fit is compromised. Incomplete polymerization leaves residual monomer which are toxic and act as an irritant to oral tissues and compromises the strength of the denture. Processing by water-bath methods results also in large numbers of voids in custom made dental devices such as a denture, which further weaken them. Improvements in the properties can be obtained by various processing methods. Examples include intensive visible-light exposure, pressurized injection water-bath heat curing and high intensity microwave irradiation. Despite improvements, resulting polymerized prosthesis are less than satisfactory and varying degrees of micro-shrinkage and porosities are present. Dentures cured by commercial microwave oven have improved mechanical properties, and have often better adaptation than those cured by the water-bath method. An additional advantage of microwave curing includes reduced processing time. Even with these advantages, dentures cured by existing microwave processing methods leads to deformations due to the large micro-shrinkage of current polymers, leading to fitting inaccuracy and unreliability. Processing porosities in the denture are not eliminated by existing microwave high power curing methods.

In the conventional thermal curing method, a temperature differential is required to force heat by conduction from the surface to the center, heat penetrates from the outside to the internal portions of the material by thermal conduction, overheating and degrading polymers at the surface. In the micro-wave curing method the object can be heated uniformly as the electromagnetic radiation instantaneously penetrates deeply and heating occurs throughout all three dimensions of the irradiated substance. The main advantages provided by microwave energy include a rapid internal heating, independent of the heat flow through the surface, resulting in a reduction of the distortion of the denture base, compared with water-bath curing and also minimal thermal lag and thermal gradients, which result in a more homogeneous cure and better properties. Additionally, high temperatures generated in the targeted object by microwave heating with available ovens and manufacturers recommendations (3 min., 550 w G.C. Acron) conduct to the thermal degradation of many thermosetting materials since very high temperatures are generated during the hardening process. Irregularity between available microwave ovens, and insufficient control of irradiation power limits the use of microwave for dental device processing.

Another example of polymers used in the dental arts is soft liners. A permanent soft liner is placed on the interface between the interior surfaces of the denture and the denture-bearing mucosa of the patients. This soft-liner should be permanently resilient, highly stable in dimension, adhering to the denture-base polymer, biocompatible, easy to clean and not capable of sustaining microbial growth. Several kinds of soft liners including polysiloxane, polyurethanes, plasticized polymethacrylates, polyvinyl chlorides and polyphosphazene fluoroelastromers are currently employed. Most soft-liners do not fulfill the above requirements due to inherent disadvantages. These include the leaching of potentially harmful bonding agents, such as epoxy and urethane adhesives, sulfuric, perfluoroacetic acid; poor adhesion to the polymethylmethacrylate (PMMA) denture base material; porosity in denture base and the liner resulting from vaporization of the solvent; dimensional changes caused by micro-shrinkage, or dehydration and rehydration steps.

The improvements of denture soft liners may be based on the use of novel materials, such as methacryloxy polydimethylsiloxanes or methacryloxyalkyl-terminated polydialkylsiloxanes.

A way to improve the fit of existing dentures is to renew its base part. This procedure which is usually done in a dental laboratory, is deficient in a manner similar to the materials that undergo water-bath hardening. Intra-oral relines can be made using chemically-cured or light-cured polymers including polymethylmethacrylates, resulting devices have a relatively low degree of cure, are porous since no compression of the dental composition is possible and often chemical and physical irritation of the oral tissues are caused.

Polymer matrix-composites are an alternative to mercury-containing dental amalgam as a aesthetic restorative biocompatible material. Composites are based on hardened polymerizable polyfunctional methacrylates used alone or as mixture with monomethacrylates, cure initiators pigments and fillers in a mixture with various comonomers such as triethyleneglycol dimethacrylate. The fillers generally consist mainly of pyrogenically produced microdispersed silicon dioxide particles.

The half-life of polymer-matrix composites cured by light is on the order of 5-8 years. The main deficiencies of composite resins include their surface degradation which leads to inadequate wear resistance, polymerization shrinkage and a lack of density. Improving the degree of polymerization is generally considered to be one way of improving composite resin performance as it would lead to stronger composite fillings and prosthodontic devices which are less susceptible to degradation, wear and fracture. It would also lead to improved biocompatibility, since there would be reduced amounts of uncured monomer to act as a biohazard. Shrinkage produces interfacial gaps, which results in microleakage, the result being bacterial penetration into the tooth. This causes adverse reactions of the pulp, sensitivity, possible pulpal death, and loss of adhesion.

The microwave curing of dental compositions under compression while they are fluid is one way of reducing problems related to the polymerization shrinkage and porosity. Microwave curing of composites while injected into a mold further reduces porosity, and enhances density, and consequently improves the biofunctionality and the durability of the dental restoration. The physical, mechanical and bio-chemical properties of a composite include strength, stiffness, hardness, abrasion resistance, toughness, ideally as similar as possible to the natural tissues. Most properties are derived from all three basic components of the composite, although some are associated with one of the three constituents. Micro-shrinkage, one of the main shortcomings of composites, is primarily due to the resin matrix. The physical and mechanical properties, such hardness, stiffness and abrasion resistance and strength, are highly influenced by resin matrix when the fillers and coupling agents are fixed. Micro-shrinkage results from the shorter distance between atoms in the resin matrix after polymerization. The monomers in the resin matrix are located at Van der Waals distance, which becomes a covalent bond distance in the corresponding polymer. Commercial resin composites are found to undergo volume shrinkage of about 1-7% with most resin composites shrinking 2-3% . Large stresses are built into the composite by micro-shrinkage, which results in adhesive failure and cohesive failure of the composite dental restoration. The micro-shrinkage also cause volumetric dimensional change which produces poor fitting to residual oral tissues. Improvements in resin formulation involve, for example, the introduction of spiro orthocarbonates and the stereoisomers. The increase of degree of conversion will generally result in the improved mechanical properties. Another problem caused by the residual monomers in the composite is the leaching of the unbound materials. The leaching has an impact on both the structural stability and biocompatibility. The residual monomers are eluted into salivary fluids and brought into contact with mucosal tissues; or be extracted into dentin and diffused to pulp. The elution decreases with the higher degree of conversion. An increase of degree of conversion will result in improved mechanical properties and biocompatibility of dental cured composites.

The principal method for treating dental caries involves its surgical excision, using various hand-held instrumentation. Carious tooth tissue consists of demineralized and softened enamel or dentin, and contains micro-organisms. Part of the infected tissue recalcify on the condition that it is kept under aseptic conditions. Infected tooth tissue which is not kept under aseptic conditions will remain as an active carious lesion, and will continue to cause progressive and destructive loss of tooth tissue. The recurrence of caries is primarily caused by microorganisms, which recolonizes a sit. In order to slow down infection and promote recalcification, it is necessary to destroy microorganisms in the carious region.

The constant exposure of dental person and devices to saliva and blood is a contributor to the transmission of infection. Available methods of sterilization have drawbacks in relation to dental devices including plastic and sharp instruments. Chemical sterilization method such as Alkaline glutaraldehyde 2% solution requires 10 hours to kill spore-forming or tuberculosis microorganisms, is irritating to human and need to be monitored since its shelf life is usually two weeks. Autoclave sterilization method expose dental objects to temperatures above 100°C for approximately 1 hour. Rubber and plastic washers and bushings within the dental handpiece and sharp edged metallic devices are damaged since heating times are long and sterilizing warm vapors are corrosive. When microwaves are used the fact that the many tools are metallic means that they will not be heated by microwaves. A second problem is the production of lighting or corona discharges between two metallic components, or at the points of instruments. Such lighting or arc melt the metal and destroy instruments undergoing sterilization. Sterilization by the indirect application of microwaves use microwaves to vaporize a liquid sterilizing solution and to expose the instruments to either the vaporized solution alone or to both microwaves and vaporized solution, a shielded pressurized atmosphere can be produced by the vaporized solution. In another microwave sterilization technique, the instrument is exposed to microwaves, e placed in a bag, drawbacks include the need to rotate the objects in three-dimensional manner and the

need to protect the oven from the energy which is not absorbed by the target and is reflected back to the microwave source requiring either an absorber of microwaves, such as water, or an absorber of microwaves within the oven which also minimize occurrence of arcing. It's also difficult to control the required temperature conditions and assure the safety of the sterilization process. One way is to surround the tools with a microwave absorbent material which will prevent the microwaves from "seeing" the tools but will become hot by itself and transfer its heat to the tools, microwave absorbing materials can also be formed into a pocket or box.

Microwave heating of materials stems from dielectric power absorption as described by the following equation: $P = KfE^2\varepsilon' \tan\delta$. The electromagnetic field energy dissipated as heat per unit volume is proportional to the dielectric loss factor ($\varepsilon' \tan \delta$) of a material, the square of the field strength (E^2) and the frequency (f) of the applied fields.

When a curable dielectric resinous material is polymerized, its microwave loss factor is drastically reduced.

Microwave power reduction and control is nearly always done by pulsing the full power on and off over some duty cycle or time base, wherein a duty cycle or time base is defined to be the amount of time from beginning the pulsing of power to the time pulsing is completed. so, for example, in an 800 watt oven, it is possible to achieve an average output of 400 watts, or 50% power, by pulsing the full 800 watts on and off, assuming the pulse width is equal to half the pulse period. These time bases are typically long, 20 seconds, full 800 watts is on for 10 or more seconds and cause lighting and hot spots problems.

One apparatus, provided in accordance with this invention, comprise a microwave applicator having a three-dimensionally defined irradiation space, including a microwave applicator having the format of a box (1), which is open at least on one side and includes a means of preventing the escape of microwave through the opening such as a door (2). The door has a means of being guided to a precise closing position such as hinges (3), and is able to be locked. The opening dimensions are preferably less than those of the walls of the cavity. The door is made of materials similar to those used for the cavity, being of good conductivity and dissipation for the electrical, thermal and microwave energy, including conductive metals or metal-plated materials. The dimensions of the cavity applicator and walls should preferably be set to minimize electromagnetic «resonance» or «standing waves» situations which may occur in some internal zones of the cavity applicator, thus causing «hot or cold spots». Therefore, the said dimensions of the cavity should not be a multiple of the wave length λg of the transmitted microwave energy or pair fractions of the said wave length such as 1/4, 1/2. For example, for the frequency of 2.45 Ghz, the wave length is: $\lambda g=n/f = 4.82$ inch.; 9.64 is a multiple of λg; 11.24 is a multiple $\lambda g/3$ and is not "resonant" and is preferred as a cavity wall dimension. A flat flange (4) made of said conductive materials is fixed to the opening of the cavity applicator, and extends outwardly from the walls, and comes into close contact with a wave trap (5), preferably mounted on the door, and which should have a dimension of $\lambda g/4$ of the emitted wave length. Leaky microwaves will be 90 degrees out of phase when going outward as well as when returning, obliging the leaky waves to travel a total of 180 degrees. The returning waves will be in counter phase with the leaking waves thus producing an energy cancellation. Each corner of the door is provided with a curved band (6) to maintain the said $\lambda g/4$ distance of the emitted wavelength, and the wave trap's efficiency. To minimize wave leakage, microwave-absorbing materials may also be installed in the wave trap. A means for efficiently locking the closed door is provided such as a T-screwing handle (7). Safety microswitches (8) are installed in a serial manner to electrically disconnect the microwave generator electrical supply when the door is open. A rectangular wave guide (9) or a cable, connects operatively the cavity applicator to the microwave source. The wave guide

includes a means of being tuned (10), and in one preferred embodiment, comprise a directional coupler (11). An aperture (12) is made both in the wave guide and in a wall of the cavity, such that they are juxtaposed. This creates a passage for electromagnetic waves to enter the cavity. The aperture preferably has a length corresponding to $\lambda g/2$ of the employed wave length and a width equivalent to the wave guide width. A deflecting plate (13) is fixed at one end of the wave guide at an angle of about 45 degrees, and causes the incident microwave beam to deviate into the cavity. The means of tuning the wave guide and system is advantageously provided on the wave guide. For example, three holes can be drill into one wall of the wave guide, and three tuning screws are placed into the threaded holes, across the said wave guide wall, the space between the holes is preferably at a distance equivalent to $\lambda g/4$. This provides an efficient means to control and reduce the standing waves in the wave guide and the microwaves which are returning to the microwave source (14).

In one embodiment a microwave the probing means consisting of the directional coupler is mounted close to the output of the microwave source on the waveguide. This coupler sense the transmitted and reflected microwave magnitude and permit the monitoring and control of irradiation parameters. The directional coupler includes high frequency detecting diodes that are mounted on a printed circuit which is mounted on the wave guide. The output of detecting diode is connected to an electronic display to permit the irradiation monitoring and control of the transmitted and reflected microwave levels through the process in real time. Preferably, the microwave probing means is connected to a central process microcontroller to follow a preset or real time self adjusted thermal processing program including irradiation modes and intensities.

In one embodiment, the control of the microwave generation is accomplished at the source by changing adequately the base voltage at the transistor, such as disclosed in diagram 1, for a precise control of the wave generator output power. For a microwave generator of 2.45 Ghz, such a magnetron, usually about -3500 DC volts are require to functions. A high voltage transformer (15)

raises the electrical voltage to about 1750 vac; then, a doubling circuit (16) composed of a high voltage condenser and a high voltage rectifying diode brings the voltage to about -3500 vdc volts. A secondary low voltage coil of 3 vac supplies the heating filament of the microwave generator. The base of the transistor (17) is connected to a micro controller (18). This transistor can be used as a variable resistor, to permit monitoring and automated management of the different irradiation and timing functions during the process. This providing permits the control of the microwave out put power in two ways. First by changing the duty cycles at the transformer which is insured by applying pulses to the base connection of the transistor. The second way of controlling of power is to reduce conveniently the applied voltage of the primary circuit of the transformer by changing adequately the base voltage of the transistor. This embodiment permits a soft management of microwave power by avoiding overheating of the microwave source, of sensitive small or high absorbency materials and corona discharge when metallic objects are used.

The generated microwave energy travels through the waveguide, is introduced and radiate into the defined exposure space from the wave guide aperture. To further reduce the standing wave patterns presence in the cavity application system, one or more microwave stirrer (19) is made with microwave deflecting blades and installed on an axle through a bushing on one or more of the cavity inside walls. The stirrer rotates by means such as a belt, pulleys and electrical motor (20). The overall surface of the stirrer can be about 3/4 of dimension of cavity's wall. Each blade have a different configuration and passes close to the aperture, causing the microwave beam to be oriented and delivered to different areas of the cavity. The materials used for the fabrication of the stirrer should have good electrical conductivity. The stirrer shaft is preferably made of a non conductive material to minimize microwave conduction and leakage through the bushing. To improve the homogeneity of the established electromagnetic fields in the cavity microwave applicator, flat or curved reflectors made of conductive, specialty materials or active electromagnetic components may be fitted in appropriate locations such as at the lower corners useful to enhance energy distribution uniformity. The apparatus is provided with a stand (21)

made of microwave transparent material to support suited dental compositions or objects that are to be microwave irradiated.

In one embodiment, in order to produce a dental polymer based object with high flexural strength and high modulus of elasticity and very low levels of post cure leachables, being preformed or not, is irradiated and internally heated while compressed by a fluid such as air or nitrogen, resting on perforated tray (22) in a flask (23) made of heat and pressure resistant microwave partially transparent materials which may be filled and reinforced such as polyester, polyethylene, polypropylene and polysulfone, and having at least two body members and a means of clamping such as screws and preferably as the disclosed bracket (24) and a pressure limiting valve (25). When used in conjunction with the provided cavity applicator, the flask is introduced in the cavity and is connected to a mechanical gas coupling means (26) being positioned on a wall and or the bottom of the cavity applicator. This permits the introduction or removal of gas as needed before, while and after the irradiation of the processed target. A gas such as air or nitrogen is introduced through one of the flask pneumatic connections such as the ring opening (27) provided with each body member of the flask and allows easy and fast processing and making of objects having highly desirable properties. Preferably, a means of rotary mechanical gas coupling which employs an electrical motor (28), permit more uniform microwave exposure of the substance or object by entertaining the flask and targeted object in a rotary movement in the cavity while under pressure a constant. Microwave absorbing substances such as water can be introduced into the flask recess to increase heat or steam generation.

In one embodiment, a means for vacuum forming method is characterized by the use of the ring opening of the lower body member of the flask and a mechanical gas coupler which is positioned at the bottom of the cavity wall, in connection with a vacuum source to allow the thermal conditioning of thermoplastic softening compositions as well as the cure of thermosetting dental material compositions with highly desirable qualities useful in many dental applications, such as

fabrication of dentures, trays or base plates, by attracting with suction the polymer-based material before and/or during the irradiation towards the mold, positioned on a dental model and a perforated tray to condition thermoplastic or thermohardening dental materials.

In one embodiment, the lower half of the flask is connected by providing coupling means to a vacuum source. A pasty polymer-based material (29) is set on or in a mold or pattern and positioned on a perforated tray in the flask. A flexible membrane (30), made of a material partially transparent to microwaves, such as silicone rubber is firmly retained by a means such as a recess between the two body. members of the flask, permitting the forming of a dental material by applying hydrostatic forces while microwave irradiated. Additional pressure can can be exerted on the dental material by the introduction of pressurized gas from the upper ring opening of the flask. The embodiment is useful in the fabrication of dental devices such as tray, base plate, fiber reinforced composite crown and bridge, and molding of thermoplastic based objects such as vinyl esther oral protectors, permitting to reduce substantially the size and the number of the voids.

In one embodiment, a dental model (31) made of materials such as wax or elastomer, which can bear components such as artificial teeth, having the forms of the object to be produced is vested in a coating material (32) such as plaster in a flask having at least two bodies members and a clamping means. First, the cup shaped recess of the lower member of the flask is filled with the coating material, then the model which may include a plaster cast is positioned in the coating material to a depth that is about half of its total height or to its largest contour. Once the coating material is set, a separating medium such as alginate based isolation solution for plaster is applied to it's exposed surface. The two parts of the flask are then joined by a retaining and alignment setup such as screws and nuts and preferably clamping bracket means. The jointly clamped body members of the flask are then filled with more fluid coating material through its upper ring opening. Each ring opening (33) can be secured to the flask by mean of threading or a shoulder. Once the added coating or mold making material is set, for dental patterns made of wax or such,

the complete flask is heated in the apparatus or in a hot water bath a few minutes to soften and melt the wax. Subsequently, the flask is split opened after removal of the clamping mean, thus exposing the internal forms of the mold and defining the shape of the object to be produced as well as holding in position, objects such as artificial teeth. All the parts are then washed with hot water. When using thermosetting material, an isolation medium is applied to all exposed surfaces of the mold to prevent the adhesion of the polymer material when in close contact with the mold at the processing stage. The fabrication method of the mold resembles to the known technique of lost wax. A drying treatment of the plaster molds can be done by its irradiation and heating in the cavity applicator or an oven. Once the dental mold is made, it's packed and may also be painted or sprayed by a dental material composition, the flask members are then clamped, introduced and mechanically connected in the cavity applicator to a fluid under pressure such as air and get microwave processed.

In one embodiment, the flask is provided with an opening (34) preferably with the disclosed means of quick connection permitting the positioning and removal of the injection nozzle (35) while flask body members are joined. The mold space within the flask is operatively connected to the flask opening through vested runners made of material such as wax, preferably set on the model before the second filling of the flask of the coating material. Physical changes, including the progressive mold filling densification and the volumetric shrinkage of many thermally conditioned polymer based material, is substantially compensated in this invention with the pressurized and when needed introduction of the fluid polymer based dental materials into the flask. The material injection means includes the use of a fluid conduct (36) with a male mechanical hydraulic coupler which allows the introduction of a fluid into the pressure injection capsule (37) through the mechanical coupler into the cavity applicator, which results in the compression filling of the materials contained in fluid dental material reservoir (38) into the mold. When under a hydraulic pressure, the piston (39) forces the material from its compartment through the injector (40) and the opening of the flask and (than) the mold is filled. The cover (41) of the

capsule is made to be removable by a means such as a threads and is connected to one or more injectors in connection with the flask. The capsule can be advantageously trained into a rotary motion transmission by means such as a key path (42), on a rotary platform pin (43) in the cavity to enhance irradiation uniformity of the mold and dental composition while submitted to hydraulic forces. The piston and the capsule are advantageously equipped with sealing joints (44). For the processing of some materials such as thermohardening polymers, the said capsule, conduct, and nozzle are preferably shielded by being made of microwave impervious materials such as steel and conserve the unprocessed material compositions in its original temperature and fluidity condition, under pressure and while being continuously available and able to be introduced as needed in the mold to compensate for the volumetric shrinkage and to fill voids and/or compensate for progressively occurring deformations of the object in thermal process. This continual pressurized injection allows a substantial increase of the dimensional precision of the produced dental objects. The presence of porosity is significantly reduced and produced objects are more suited for dental uses in terms of biofunctionality, fit and durability when compared to objects such as prosthetics, produced by the conventional methods and materials. Preferably, a bleeder (45) made of microwave transparent materials is employed in an appropriate housing made on at least one of the flask members closing surface, and provides a means of hydraulically connecting the mold to the exterior of the flask, useful in reducing the energy and time required to appropriately fill the mold and also minimize porosity occurrence. Said bleeder accelerate the emptying of the existing air of the mold space when introducing resinous materials into it while preventing the leakage of resinous fluid dental materials under pressure by moving outwardly and blocking the external orifice of housing.

In one embodiment, low microwave absorbing materials such as thermoplastic resins are indirectly heated with the use of a compression-injection capsule coated or layered with microwave absorbing substances such as metal oxides including zinc oxide, carbon black and ceramics.

In one embodiment, an economic manual fluid resin pressurization and injection device (46) is provided to remove the need of being connected to an external pressurized fluid source. A mechanical force accumulator such as a spring (47) is compressed by turning the internally threaded cylinder (48) while holding the device handel (49). A force boosting piston (50) is especially useful for molding and filling of composite curable dental materials. The injection nozzle and the piston acts as previously described. This embodiment can be used with the disclosed cavity applicator and flask or with the mobile microwave applicator.

In one embodiment, a shielded temperature probe such as a thermocouple made with a temperature dependent resistor, a fluoro-optic or an infrared temperature magnitude detecting means is used with optionally a pivoting electrical connector to permit the sensing of the thermal conditions of the microwave irradiated target. This embodiment permits a precise setting of the pace of thermal conditioning as well as the indication of the reach of a specific temperature magnitude, useful for the thermal processing of delicate materials such thermoplastics or low temperature boiling monomers as well as to increase safety in the sterilization functions and is preferably used in connection with the central micro controller.

In one embodiment of the present invention, to permit a safe and quick sterilization of dental objects without fear corrosion or arc occurrence, a cylindrical column (51), made of microwave transparent materials is closed at one end and externally threaded at the neck, made sufficiently thick glass or polymer to resist heat and pressure, in used in conjunction with the provided flask and cavity applicator. The cylindrical column permits heating of a liquid and hot steam generation and optionally the production of a microwave shielding atmosphere is screwed into the lower flask half member through it's ring opening with its sealing joints (52). A liquid such as distilled water is introduced to fill the column, up to a pre-determined level. A specially shielded flask operatively connected or not with the steam generation column is introduced in the said cavity

through the door or only its column introduced from the provided top circular opening (53) into the cavity applicator which is provided with a disk form closing door. To sterilize, the steam having reached the evaporation temperature under microwave irradiation fills the flask with the vapor rise up. The upper flask half is preferably made of a heat conduction and exchanging material (54) such as stainless steel and comprise a heat sink to cool by conduction the internally contacting warm vapor. The condensed and liquefied sterilizing solution return by gravity to the base of the column where it's repeatedly heated and evaporated, providing a constant steam flow and contact with treated dental objects contained in the flask. To detect the temperature magnitude with high accuracy, the temperature probe for microwave environment can be placed within the flask. The flask can be sealed immediately following removal of the probe after sterilization with the use of an annular elastic sealing coupler positioned on one of the flask inlets such as the injection opening or the pressure limiting valve manifold. The means of microwave and temperature magnitude detection permits a precise control and delivery of microwave to a dental target, useful in avoiding arcing occurrence by generating adequate microwave power levels and/or creating a shielding vapor pressure atmosphere inside the flask. The temperature and microwave sensing and control is preferably done in an automated manner with the programmable micro controller. Once the predetermined temperature is reached, a signal is sent to the microcontroller which then reduce the power of emission so as to maintain a sufficient amount of time to sterilize (6 min). Equilibrium temperature is reached quickly since there is no great swings in the temperature and optimal control of the microwave delivery is achieved.

In one embodiment, the temperature is safely and economically controlled for sterilization function through a gas pressure sensor which is connected to the flask for example through the pressure limiter manifold (55) or vent to control the sterilization temperature inside the flask, specially when used with the shielded flask, positioned externally with only its steam column introduced in the cavity applicator. This pressure sensor is operatively connected to a microcontroller to maintain the right warm steam pressure temperature magnitude and permit

monitoring. The temperature sensor for microwave environment can also alone or jointly with the pressure sensor be used with the disclosed device. Any increase of temperature of a gas having a given volume conduct to an increase of it's pressure. By limiting and/or controlling the pressure of the gas, an effective control of flask internal temperature is achieved. The micro controller control the flask internal temperature via the microwave generator, using the provided microwave power control.

We have conducted complex dielectric permittivity, temperature and distribution pattern studies of microwave heated teeth and Simulations of specific absorption rate distribution. The complex permittivity was measured on different types of dental tissues, using extracted teeth, including enamel, dentin and caries. Reflective coefficients has been obtained using a network analyzer. The characteristics of enamel caries and dentin are different. Some caries are soft. The higher the moisture of caries, the higher is its permittivity. The dielectric loss factor of caries is fairly higher than that of normal healthy parts. When the tooth is exposed to microwaves, caries are preferentially heated. Temperature rise can kill the microorganisms in caries. Control and or extinction of microorganism slows or stops the progress of caries, permitting previously carious tissue to recalcify by biological latent support of the pulp. Temperature distribution measurement with microwaves heating reveals that the temperature of caries is higher than that of normal tooth tissue. These properties are used with the provisions of this invention for the diagnostic and treatment of teeth having caries and subsequent internal heat conditioning and or curing of provided dental restorative materials. When dielectric loss factor is higher, the absorption of microwave is better and local temperature is higher. Microwave energy heats by radiation and is able to penetrate through various substances including desiccated tissue and thus can create an addressed effect.

In one embodiment, a method of caries control in a non invasive atraumatic way, without surgical burs entry and with a reduced risk and necessity of exposing the dental pulp organ comprise, a

hand held microwave applicator with an electronic circuit, (diagram 2) designed to suit a small microwave generators such as impatt diodes with an output power of about 5 watts which requires usually an electrical voltage of about 60 DC. The electrical current is applied through a high impedance line (56) in order to limit the perturbation of electromagnetic signals. The power supply module is provided with a current and voltage limiting mean to permit the polarization of the impatt diode in the specific limits with a resonant circuit (57), such as a 50 ohms line, having a length preferably equal to the half of the length of the selected frequency. The length of the line may be calculated with the following equation: $L= 3x10^8 / 2F_r\, \varepsilon_{cff}^{1/2}$. One end of the "resonator" is connected to the impatt diode (58) and the other end of it is coupled (59) to a transmission line including an isolator (60) to provide isolation of the microwave source from the rest of the circuit in order to avoid frequency variations, caused by a mismatch of the output (61). A coupler (62) having a coupling of about -15 dB permit a sampling of the signal emitted by the microwave generator in order to measure the forwarded and reflected power. The couplers should be perfectly matched at both extremities to permit precise measurements. Matching circuits (63) at the input and the output as well as load resistors permit achievement of an adaptation at each ends, equal or better than -15dB. Detecting diodes (64) rectify the radio frequencies signal in order to convert the power to a dc voltage which can advantageously be subsequently transmitted to a micro controller or a "ADC" which converts this voltage to a numerical signal for an appropriate processing of the acquired informations and the precise and monitoring control of microwaves energy delivery to the dental target. The said control is a means of controlling the power level, exposure cycles, processing modes, as well as the selection of the frequencies of microwave generation. The control of the microwave generation is preferably made by a selector (65), located on the device, allowing to set different power levels and modes. Between the tip antenna and the microwave source or amplifier, a shielded cable (66) or wave guide, as short as possible is used to operatively transmit the microwave power to the head antenna.

This provided method and device of controlling and treating dental caries in a non-invasive atrau-

matic therapeutical approach does not require surgical burs entry and reduces the risk and the necessity of exposing the dental pulp.

An insulated connection permits the interchange of different provided head antennas to match different applications and enhance energy transmission and deposition on the dental target. A means of electrical supply (67), such as a shielded cable, connects the mobile applicator to the power supply. The hand held applicator is preferably equipped with a water cooling system (68) and a digital display (69).

One head antenna (70) provided for therapeutic purposes to target teeth and treat and heat or detect dental caries is made of a highly conductive metal such as copper, platinum or gold, plated or not, having the format of a rectangular or a loop shaped band of which one end is connected to the internal metallic lead of the device cable and the other connected to the external metallic lead of it.

One provided head antenna has the form of an I. This applicator is a short monopole, made for example by stripping the outer jacket and the outer conductor of a coaxial shielded cable, the inner conductor and dielectric (Teflon) constitute the applicator. A loaded I-applicator (71) having an increased energy forwarding capacity is made by soldering a platinum ring over the outer conductor of the coaxial cable and soldering a platinum rod to the tip of the inner lead of the cable.

Another provided head antenna (72) is made of a microstrip being made of miscible polymeric or other conductive materials, for example, a square metal skin is positioned on a non conductive material with a ground plane on its back, particularly useful for the heating and/or joining of dental object.

In one embodiment, an electrically shielded temperature probe is embedded in the head of the

hand held applicator antenna to provide a means of monitoring the temperature of the heated target for judging the efficiency of tissue heating and to avoid sudden temperature rises.

The provided head antenna designs help in achieving good impedance matching and effective delivery of microwave for internal heat conditioning of dental targets. A means of safely containing any escaping microwave energy close to the irradiation space can be used such as the disclosed head antenna choke (73), made of microwave absorbing foamy or rubbery materials.

In one embodiment, head antennas are equipped with a miniaturized version of the membrane dental fluid material adapter in order to assist intra-oral tooth restoration.

In general, various polymer based material compositions are useful for the construction of dental devices. These compositions may be used in the filling of teeth and the construction of appliances used for replacing teeth and other oral structures. One utility of these compositions is in the construction and repair of removable dental devices such as dentures and dental anchored restorations such as crowns, bridges, inlays, veneers. Also utility is found in the making of mouth guards, oral border molding, impression trays, base plates, orthodontic dental appliances. Various thermoplastic containing dental compounds are also advantageously thermally conditioned and softened while formed with the provided method and apparatus.

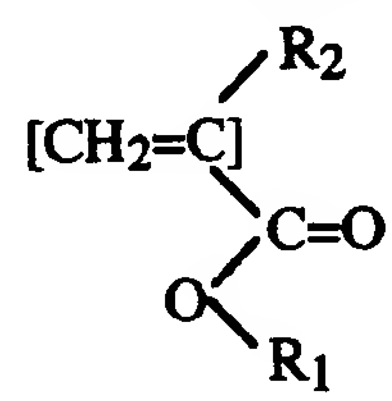
One preferred composition for dental aesthetic composite suited to be formed and hardened in accordance with the providing of this invention consists of a polymerizable mixture including one or a selection from the large family of polyfunctional methacrylate esters, and oligomers including the compound prepared from one molecule of bisphenol A and two molecules of glycidyl methacrylate called 2,2bis[4(2-hydroxy-3 methacryloyloxy-propyloxy)-phenyl] propane, known as Bis-GMA for its lower degree of shrinkage and/or 2,2-bis[4-methacryloxyethoxy)Phenyl] propane for its good water resistance properties. Other monomers, such as triethyleneglycol dimethacrylate for viscosity reduction, urethane dimethacrylates, spiro orthocarbontes are advantageously employed in admixture with silanized inorganic fillers and organic fillers,

coupling agents, microwave sensitive cure initiation system including organic peroxides and amines and color pigments are advantageously added. The weight of the fillers as an overall weight of the composite is preferably in the range of 30 to 90 % and include silanized silicon dioxide particles.

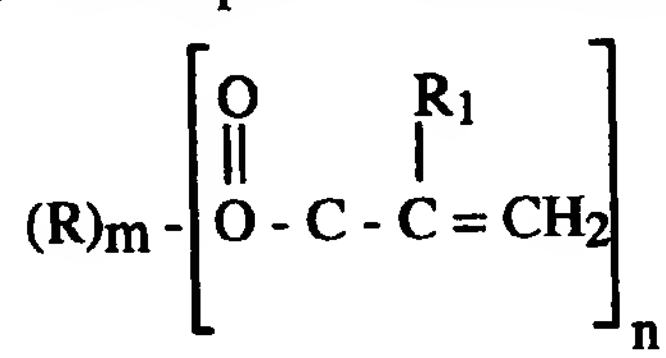
In one embodiment, compositions specially suitable for making dental removable appliances such as dentures is provided which comprise a liquid and a powdery component.

The liquid component in accordance with the invention contains preferably from 40% to 90% of mono-, di-, tri-, or multifunctional acrylic monomer, a cross-linking agent, a plasticizer, a stabilizer. an accelerator and color pigments.

The mono-, di, tri, or multifunctional acrylic monomer in accordance with the invention are within the scope of the formula:



where R1 in accordance with the invention is hydrogen, alkyl, substituted alkyl group, cyclic hydrocarbon, benzyl, ether, hydroxyalkyl and R2 is hydrogen, halogen, alkyl, substituted alkyl or cyclic hydrocarbon group. Monomers within the scope of the following formula are also particularly suitable to the invention:



wherein R is an acrylic-free organic moiety, R_1 is hydrogen, hologen, halogen, alkyl, substituted alkyl or cyano radical and n is an integer from 1 to 20 and m is an integer from 1 to 1000.

These monomers may be used alone or in admixture.

The microwave sensitive initiators in accordance with the invention includes benzoyl and peroxide, dilauroyl peroxide up to 2,5%.

The polymerization accelerator in accordance with the invention is a quaternary ammonium

chloride, which is easily soluble in the methacrylate monomers and reacts with barbituric acid derivatives. A preferred compounds are the quaternary ammonium with an alkyl of 1 to 20 carbons, such as, dodecyltrimethylammonium. These quaternary ammonium chlorides may be added in alone or in admixture from 0,09 to 1,5 %.

The crosslinking agent in accordance with the provided microwave hardening material compositions is a polyfunctional monomer wherein at least two carbon-carbon double bonds, such as 1,3-butanediol dimethacrylate, 1,4-butanediol dimethacrylate, 1,4-butanediol divinyl ether, di(ethylene glycol) dimethacrylate, di(ethylene glycol) divinyl ether, pentaerythritol diacrylate monostearate, ethylene glycol dimethacrylate, trimetylolpropane trimethacrylate, pentaerythritol triacrylate, pentaerythritol tetraacrylate, trimetylolpropane triacrylate. The crosslinking agents may be used alone or in admixture.

Polymerization promoters for the monomers of the provided curable material system including acrylates consists of a barbituric acid derivative which, when irradiated with microwaves, rapidly react with the quaternary ammonium chloride to produce radicals, which promotes a rapid and uniform polymerization in the composition and a higher degree of conversion. The barbituric acid derivative in accordance with the invention include 1,3,5-trimethylbarbituric acid, 1,3-dimethyl-5-isobutylbarbituric acid, 1,3-dimethyl-5-phenylbarbituric acid, 5-n-butylbarbituric acid, 5-ethylbarbituric acid, 1-cyclohexyl-5-ethylbarbituric acid and 1-benzyl-5-phenylbarbituric acid. These acid derivatives may be used alone or in admixture in very small amounts.

The polymerization stabilizers comprise hydroquinone, hydroquinone monomethyl ether or 4-ethoxyphenol which are usually added to the liquid component of dental compositions (up to 4%).

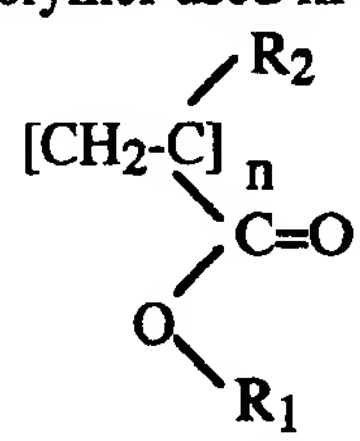
The plasticizer in accordance with the invention is generally a low molecular weight ester, such as dibutyl phthalate or phosphates .

The composition for a one component microwavable curable material system in accordance with this invention is approximately the same as the one for the two component materials with some variations mainly in the initiation system. Preferred initiators for a one component dental composition for denture or such need to be thermally stable at room or higher temperatures such

as 50°C and initiate polymerization at higher temperatures such as benzopinacole, tert-butyleperbenzoate, and 2,2'dichlorobenzopinacol.

The powder component in accordance with the invention includes from 20% to 80% of mono-di-tri, or multifunctional acrylic or acrylate ester polymer. The powder may advantageously include from 5% to 40% of a copolymer. The powder component in accordance with the invention may advantageously include from 0,1 % to 3 % of an initiator for radical polymerization such as organic peroxides. The powder component in accordance with the invention can include up to 1% of a barbituric acid derivative to promote chemical reaction.

The mono-, di, tri, or multifunctional acrylic polymer used in denture base in accordance with the invention are:



where the R1 in accordance with the invention is hydrogen, alkyl, substituted alkyl group, cyclic hydrocarbon, benzyl, ether, hydroxyalkyl, R2 is hydrogen, halogen, alkyl, substituted alkyl group and n is an integer at least equal to 2.

The copolymer in accordance with this invention are mainly composed of methyl methacrylate polymer or a mixture of methyl methacrylate polymer and an methacrylate polymer other than methyl methacrylate polymer.

Inorganic and organic fillers may be added into the compositions of one or two components denture base. Useful inorganic fillers include glass, metal ceramics, silicon dioxide in powdery or fiber format, which are preferably silanized with coupling agent, such as 3-methacryloxloxypropyltrimethoxy. Organic fillers include splinter or bead polymers of high molecular weight , or fibers such as aramide fibers, polyacrylate fibers, polyamide fibers and polyacrylonitrile fibers. Organic fillers may be used alone or mixed with inorganic fillers.

Microwave curing resilient compounds for making devices such as denture liners is molded and

cured with the provided novel method and apparatus including organopolysiloxanes and phosphonitrilic fluoroelastomers [poly(fluroalkoxy)phosphazene] with a crosslinking agent, a filler and an initiator. Silicones are containing a repeating silicone-oxygen backbone with organic side groups attached via carbon silicone bonds. One composition for soft denture liners in accordance with this invention contain silicones within the scope of the structural formula:

$$\left[R_nSiO_{(4-n/2)}\right]_m$$

Wherein n=1-3 and m>1. R groups are usually methyl, longer alkyl, fluoroalkyl, phenyl, vinyl, alkoxy or alkylamino. One preferred silicone compound is polydimethylsiloxane (PDMS) of the following structure:

$$-\!\!\left(\begin{array}{c}CH_3\\|\\Si\\|\\CH_3\end{array}-O-\begin{array}{c}CH_3\\|\\Si\\|\\CH_3\end{array}-O-\begin{array}{c}CH_3\\|\\Si\\|\\CH_3\end{array}-O\right)\!\!-$$

Methacryloxy-terminated polydimethylsiloxanes are particularly useful since they bond well to PMMA made dentures due to the chemical similarity.

The crosslinking agents for soft liners are normal multifunctional monomers wherein at least two carbon-carbon double bonds. Preferred crosslinking monomers are acryloxy or methacryloxyalkyl-terminated siloxane monomers, such as 1,3-bis[(p-acryloxymethy) phenethyl] tetramethyldisiloxane, 1,3-bis(3-methacryloxypropyl) tetramethyldisiloxane, due to chemical similarity.

The normal initiators in the soft denture liners in accordance with the invention are general peroxides, such as benzoyl peroxide, lauroyl peroxide, which are usually added to the powdery component of resilient compositions in small amounts.

The phosphonitrilic fluoroelastomers (poly(fluoroalkoxy)phosphazenes) in accordance with this invention are polymerized by monomers within the following formula:

$$\left[N{=}P\begin{array}{l}\diagup OCH_2(CF)_{2n}X\\ \diagdown OCH_2(CF)_{2n}X\end{array}\right]$$

wherein X is H or F, and n is usually from 1 to 11, 30 to 60 % of total ingredients for a firmer liner

and up to 90 % for softer one.

The crosslinking agent suitable for the fluoroelastomers are monomers with at least two functional groups, such as tetraethylene glycol dimethacrylate, ethylene glycol dimethacrylate, 1,6-hexamethylene glycol dimethacrylate, trimethylopropane trimethacrylate, pentaerythritol triacrylate, pentaerythritol triallyl ether, pentaerythritol tetraacrylate.

The fillers,which are preferably mainly hydrophobic improve hardness and the ability to grind and polish the cured resilient materials and the bond durability between the liner and base. Particles of fillers may be beads or fibers, pigments and other additives can be added to the soft material system (fillers 7 % for soft, 30 % for firm liners).

Thermoplastic compounds such as poly functional methacrylate, polycarbonate, polysulfone, fluoropolymers, elastomers, polyurethanes, impression compound, wax, polycapratone and mixture of thermoset and thermoplastics are advantageously heat processed with the provided method and permit dental rehabilitation.

Microwave absorbing substances can advantageously be incorporated into disclosed thermoplastic and thermohardening material compositions, to decrease internal heat generation of compositions which does not have sufficient dielectrical loss when microwaved nor does they have sufficient heatability for a desired speed of heating. These microwave «absorbers» are also useful when the employed polymeric material has only a low microwave absorption behavior at low temperatures such as many thermoplastic polymers including polycarbonate and also for substantially increasing the speed and the adressability such as in welding and joining functions. These absorbers may be powdery, hollowed, coated and comprise ferromagnetics, metallic oxides or speciality ceramics.

All ratio for materials are expressed in weight.

Examples

Cavity applicator dimensions
- Cavity: 32 cm x 32 cm x 28 cm made of steel
- Wave guide: 3.8 cm x 7.6 cm x 45.7 cm such as WR 284 made of copper
- Steerer: 20 cm made of steel

Flask (made of polypropylen)
- diameter interior: 8 cm
- diameter exterior: 13 cm
- recess depth: 1.5 cm
- bleeder 2 mm diameter 3.5 mm long
- membrane thichness: 3 mm
- ring: 3.5 cm

Injection capsule dimensions (made of stainless steel / wall thickness 6 mm)

	Diameter	Stroke	Piston height
- Dentures:	10 cm	5 cm	2 cm
- Manual:	5 cm	6 cm	2 cm
- Composite boosting piston:	3.5 cm	2.5 cm	1.5 cm

Process programmable micro controlle
Micro-controller Pic of Microship inc. or Parallax

Microwave frequency
- Magnetron frequency: 2,45 GHz Output power 600 W
- Impatt diode frequency: 24 GHz Output power 5W

- Vacuum source such as an 600W cleaning aspirator for dental
  Vacuum forming of resinous or microwave softened dental materials
- The steam generation column is made of polycarbonate with it walls having a tickness of 1 cm, 6 cm inside diameter and a height of 12 cm

Pressure limiting valve
- Aperture: 4 mm^2
- Weight: 80 g
- Pressure: 24 PSI

Sterilization method	**Rapid heat & steam generation**	**Warm steam maintenance**
Water 200 cc sterilization equiped flask with only its column introduced in the cavity. Pressure limiting valve set a 25 PSI, 80 g	450 W, 2 min, water is brought to the boiling temperature	Max 25 PSI, temperature 100° C 125 W, 5.5 min

Experiment of decay control in the cavity microwave applicator

	Preparation	**Microwave irradiation**	**Incubation**	**Results**
Section of decayous freshly extracted human teeth prepared, 2 mm^3	Surface desinection, 15 seconds deeping in cloramine T solution	1.5 W/cm^2 energy density of irradiation (200 W in the cavity application) 60 sec.	Culture of irradiated decayous teeth section in medium at 37°C 24 h.	Delivered rradiation destroy completely carious zone microorganisms

Examples of microwave procressing of polymer based material	**Steps of the procedure in order**		
	Compression, forming	**Microwave irradiation**	**Bench cooling**
Aesthetic composite			
A 100 de 3M inc, color ivory, 0.15% of benzoide proxide for initiation. 1cc Example 1	1 min	3 min, 450 W	3 min
* Example 2	2 min	5 min, 250 W	3 min
* Mechanical test (3 points bend, failure) of specimens of example 2	Size: 25 x 2 x 1.75 mm	Load at max	Displacement at max
Testing spécifications, crosshead speed 2 mmm/min, Instron device	25 PSI membrane compression , flask &plaster mold	45 N	0.42 mm
	Mold injection filling	**Microwave irradiation**	**Bench cooling**
GC Acron resine for dentures , 40 cc Flask with bleeder plaster mold Large capsule Example 1	100 psi,3 min	7 min,. 225 W + 1 min, 400 W	6 min
Example 2	100 psi, 3 min	4 min 450 W	6 min
Soft materials Molloplast B, Regnesi & co GER, 40 cc	80 psi, 5 min	12 min ,225W + 1.5 min, 400 W	6 min

Repair, soldering of denture resin

G-C ACRON, denture repair material powder & fluid	~ 25 psi, air pressure - 80 g pressure limiting valve weight on a regular dental index made of plaster 2 min, 200 W + 1 min, 350 W

Microwave softening of thermoplastic dental material	**Microwave irradiation**	**Adaptation time**
Border molding coumpound in a 5 cc seringue	4 min, 200 W	2 min
Dental custom tray, polycapratone sheet thermosoftening Thickness: 3 mm	2 min, 300 W	1.5 min

FIG.1

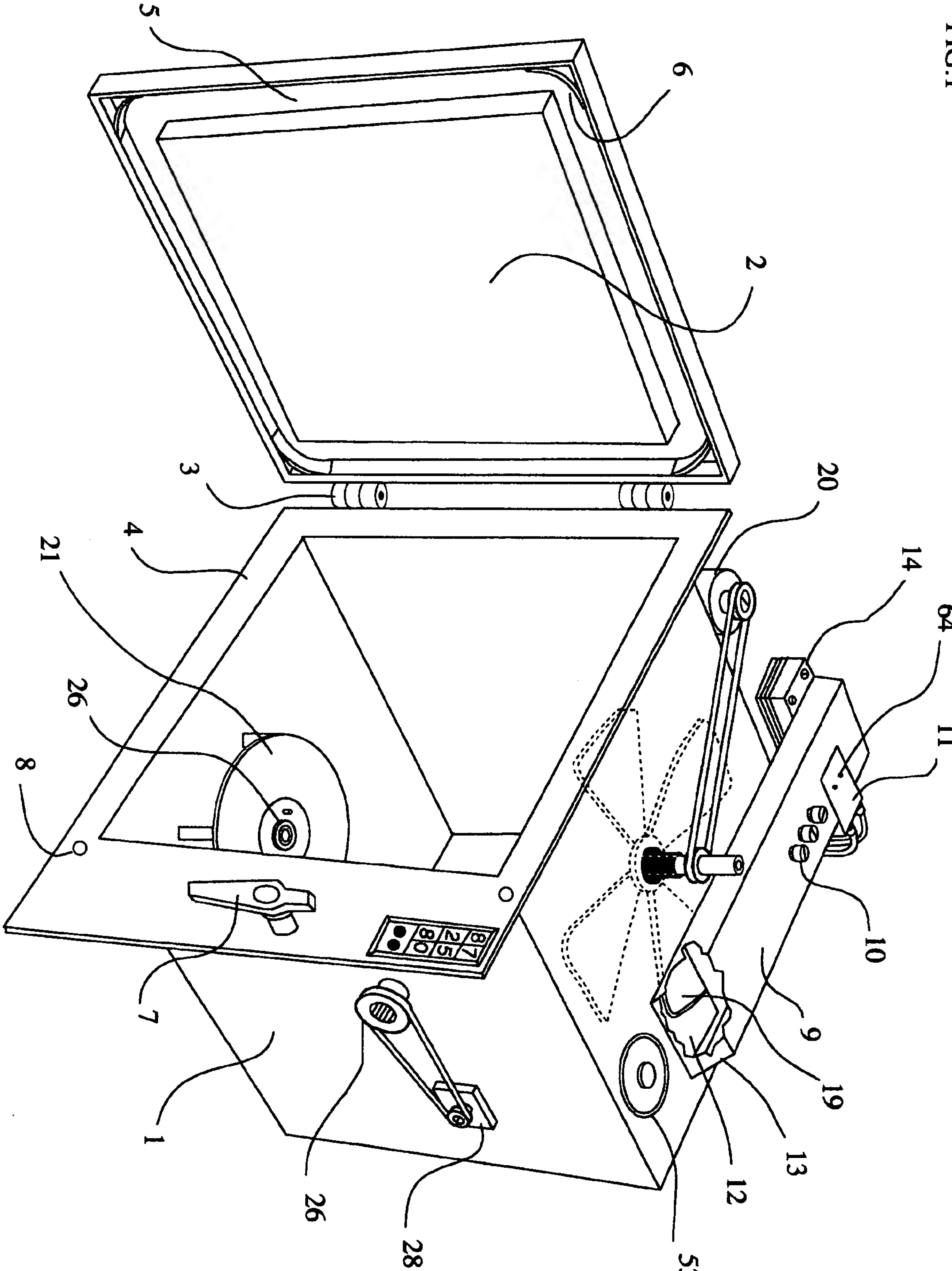


DIAGRAM 1

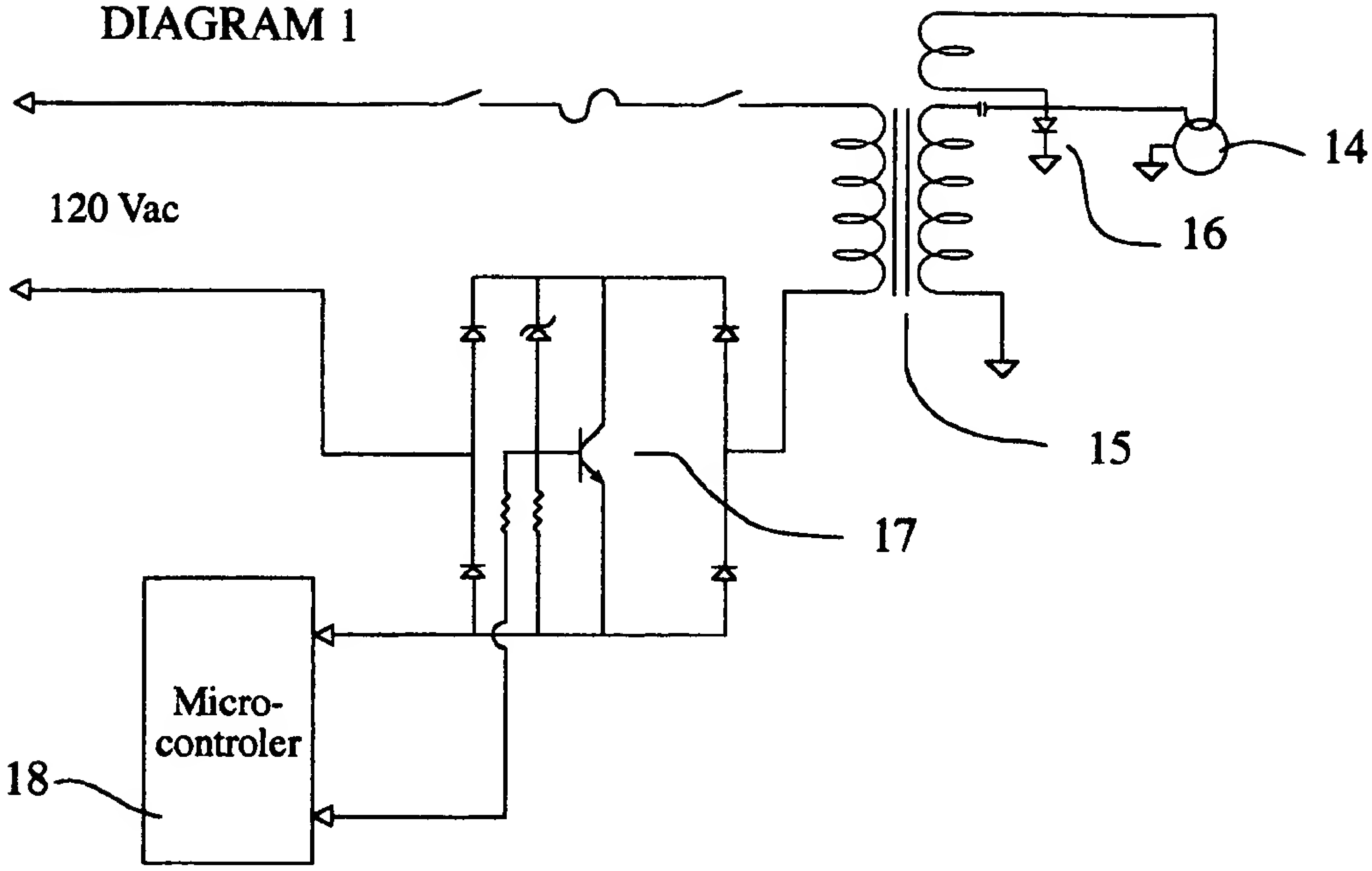
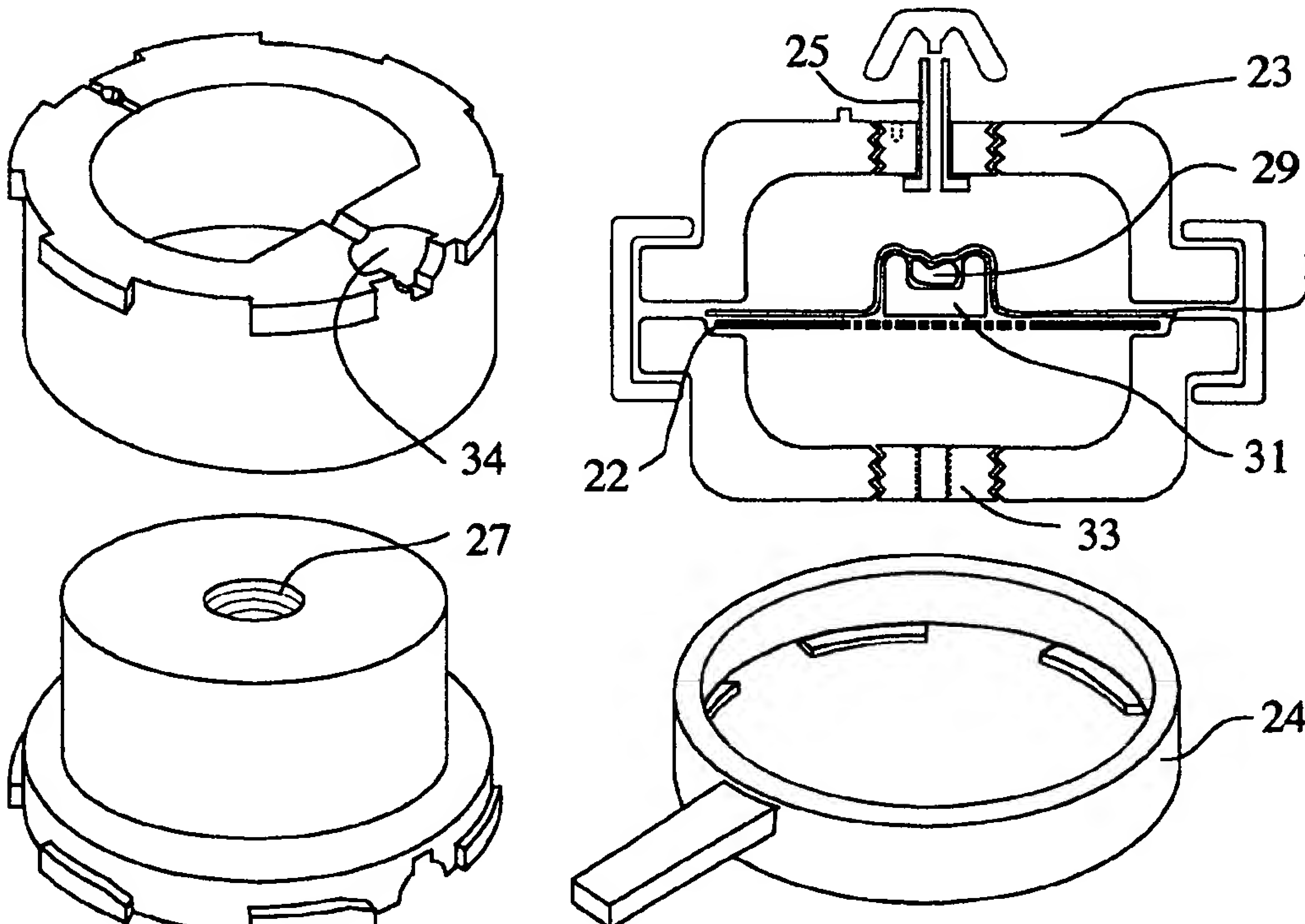


FIG. 2



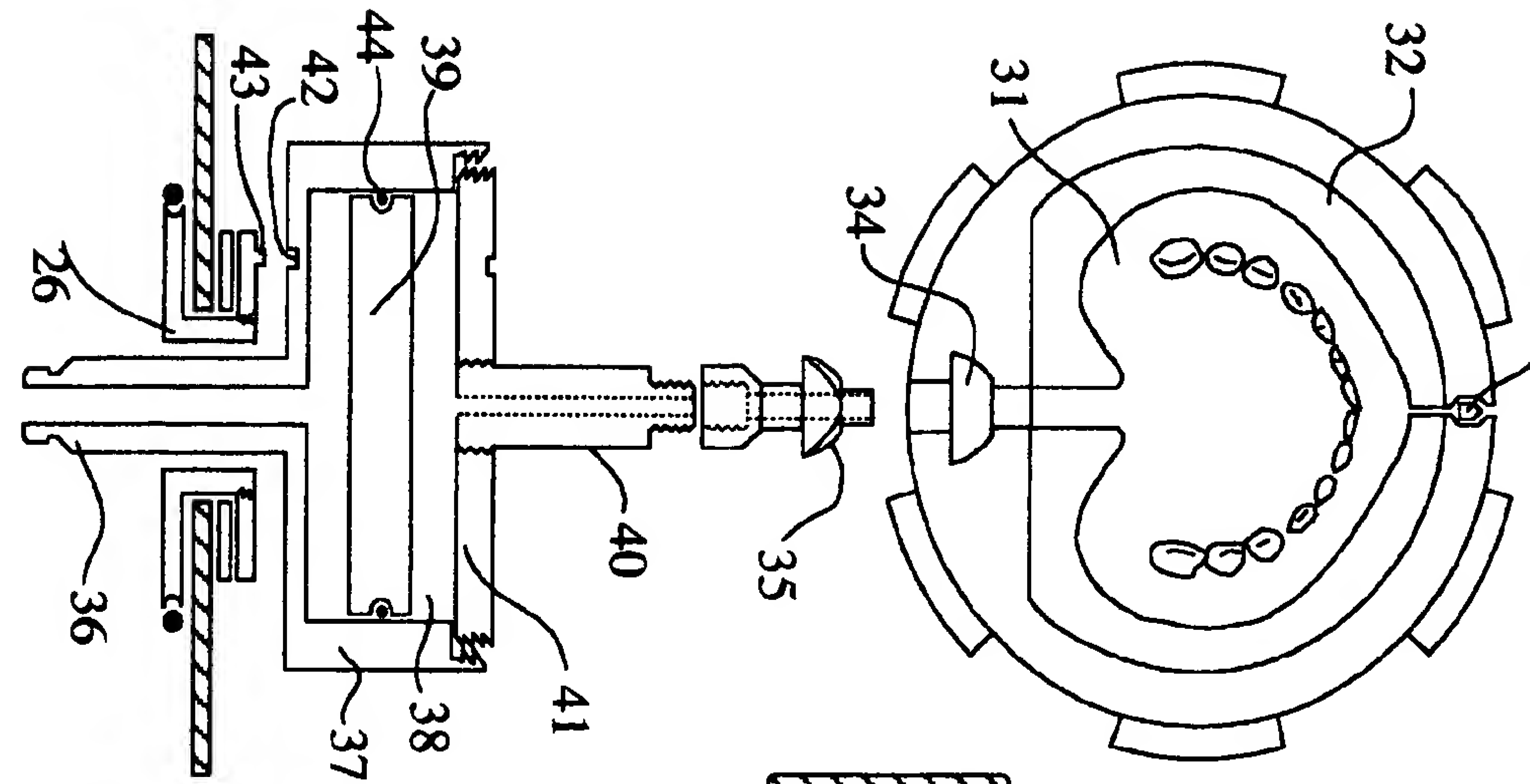


FIG. 3

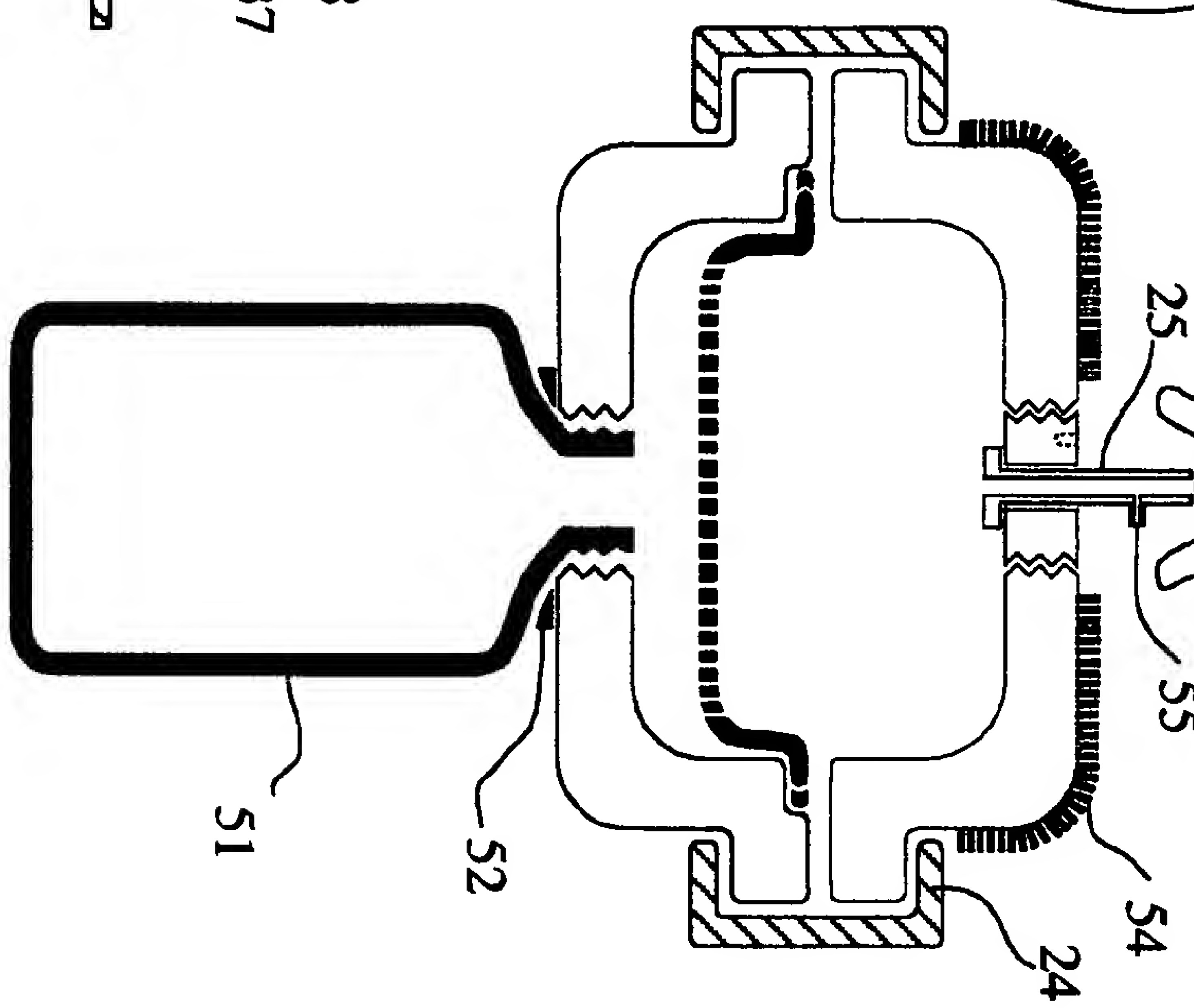


FIG. 4

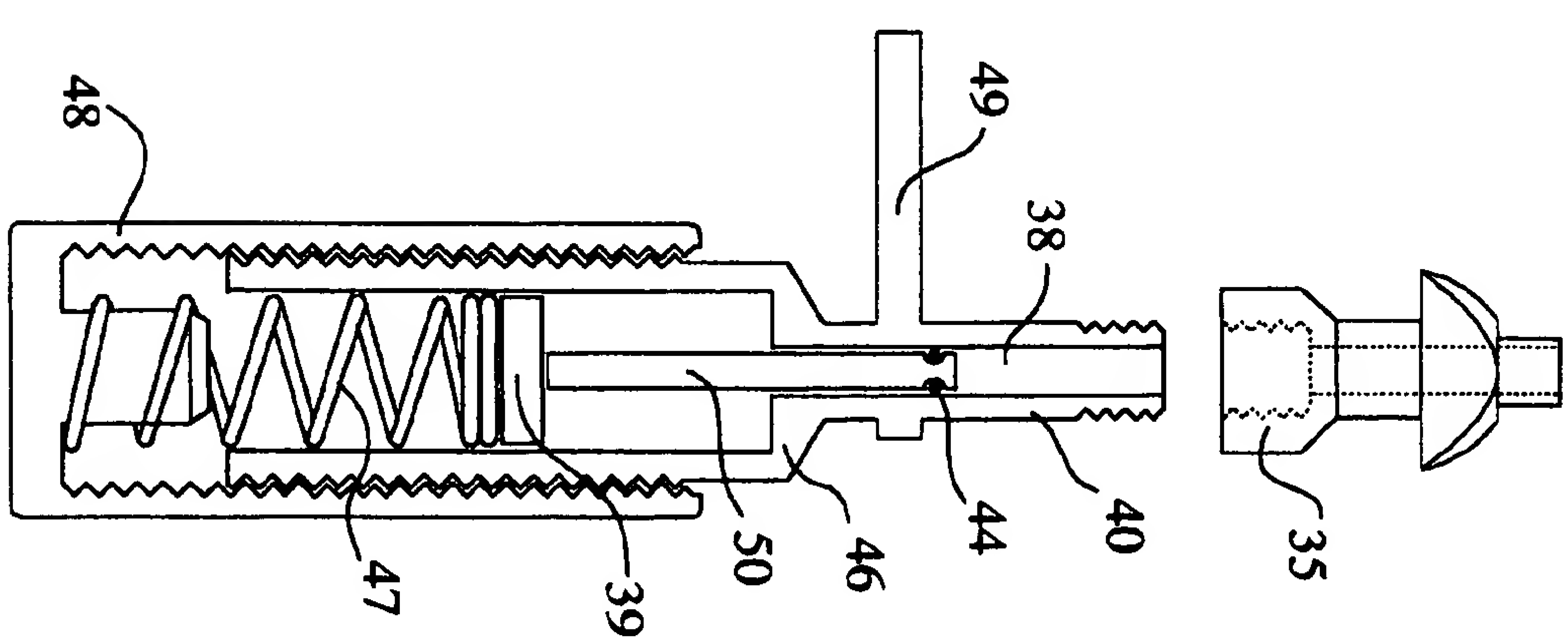


FIG. 5

FIG. 6

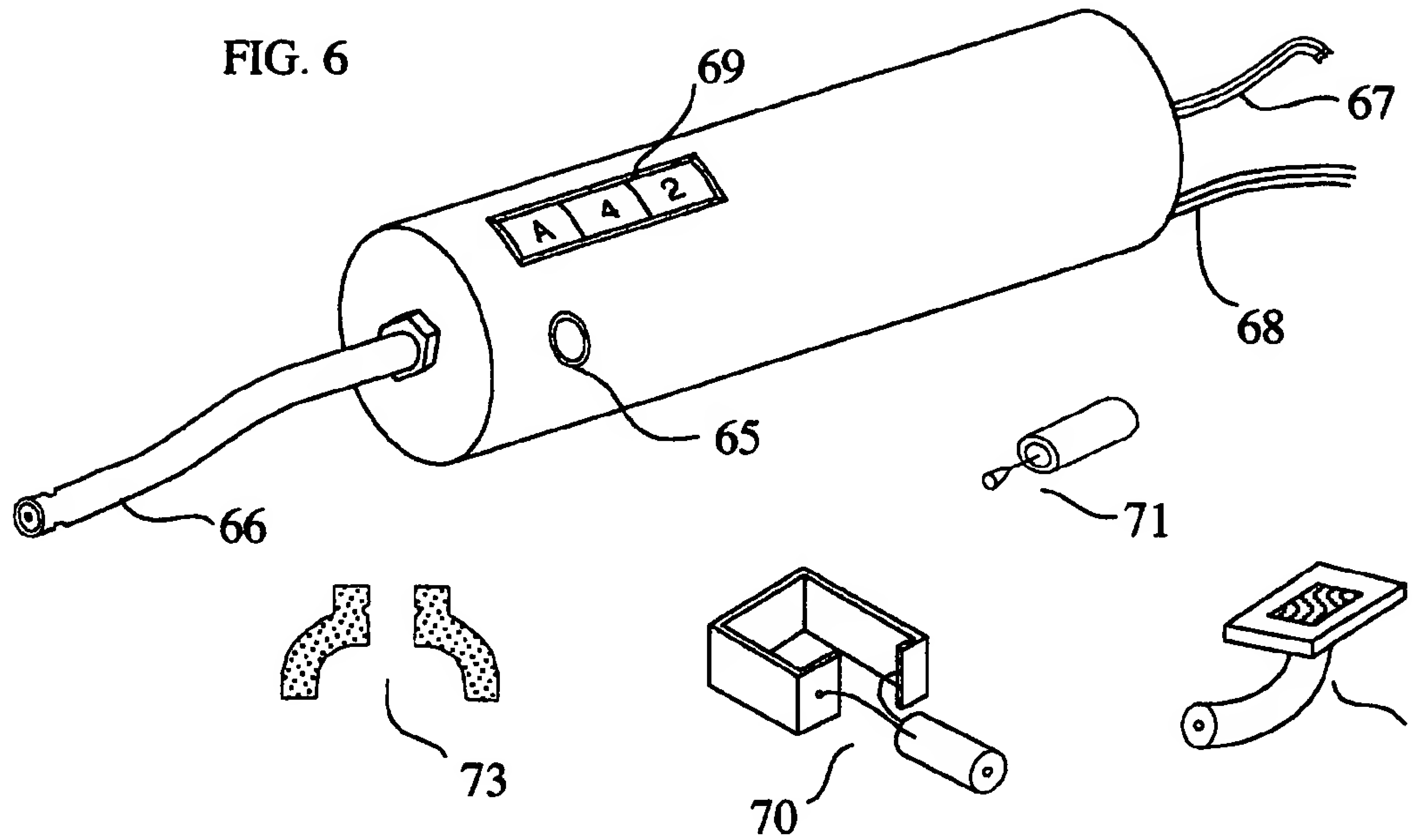


DIAGRAM 2